

MAPLE SIRUP. VIII. A SIMPLE AND RAPID TEST FOR THE ANALYSIS OF MAPLE SIRUP FOR INVERT SUGAR^{a, b}

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A simple, rapid threshold test for the approximate analysis of invert sugar in maple sirup is described. The method is based on the selection of sample dilutions such that, for any given percentage of invert, one dilution will give a positive color test while the adjacent higher dilution will give a negative test reaction. The test requires only a simple kit, obtainable from all drug stores, and such equipment as can be found in the home or processing plant.

Many farmer-producers of maple products extend the income from their maple crop by making and selling a number of maple confections in addition to maple sirup. One of these confections is a maple cream or "butter" (a fondant type of spread) which consists of a myriad of microscopic sugar (sucrose) crystals in sirup. The product, like many of the "butters," can be spread on bread, toast, pancakes, and other food. The process used in making this maple spread is relatively simple. Ordinary commercial sirup is boiled until sufficient water has been evaporated to give a sugar solids concentration that will elevate the boiling point approximately 20°F. above that normal for boiling water. To prevent growth of large crystals the mass is rapidly cooled to about 100°F. and then stirred, either by hand or with a mechanical mixer, until the sucrose crystallizes to a cream-like consistency.

To obtain creams of the proper consistency the amount of simple sugars (invert sugar) must be within certain limits. When no invert sugar is present and no provision is made to form it during the preparation of the product, the cream may have an undesirable, coarse, gritty crystalline structure. However, this condition seldom is encountered. On the other hand, if a large amount of invert sugar is present no crystallization of the sucrose will occur. If the amount of invert sugar is only slightly in excess of optimum the cream may be too fluid and will separate on storage, producing a product of unpleasant appearance. Just what the optimum level of invert sugar is, is governed by a number of conditions such as pH and concentration of solids (boiling point elevation). The amount of invert which can be tolerated is not critical within a range of 0.5 to 2%. Somewhat larger amounts of invert (2 to 4 per cent) can be tolerated providing the temperature of the boiling sirup is increased by 2° to 4° above the normal

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^b Maple Sirup VII was the article by Porter, W. L., Hoban, Nancy, and Willits, C. O., entitled "Contribution to the Carbohydrate Chemistry of Maple Sap and Sirup," *Food Research*, 19, 597-602 (1954).

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of 20°F. The amount that the temperature must be increased will depend on the amount of invert sugar present. This, however, necessitates that each batch of sirup must be tested in small lots (by trial and error) prior to making the large batches of cream.

A simpler procedure would be the use of maple sirup which has an invert sugar concentration which falls within limits fairly close to the optimum (0.5 to 2%) so that standard cooking temperatures and process can be used. This procedure would require a chemical analysis of the sirups. Unfortunately, most producers do not have available on the farm the facilities necessary for such determinations, and cannot afford to have the analyses made by a commercial analytical laboratory. This paper reports a simple method for an approximate analysis for invert sugar in maple sirup, and which is effective on sirups containing up to 7% of invert sugar.

TEST FOR INVERT SUGAR

The test to be described is a simple, inexpensive, chemical method of analysis which may be used to obtain an approximation within 1% of the invert content of maple sirup. It is best applied as a limiting test by which a sirup can be shown to contain more than or less than a certain percentage of invert sugar. The test is based on the use of a simple reagent^a which was developed for the determination of sugar in the urine of diabetic patients and is obtainable at any drug store. In addition to this reagent, medicine droppers, and test tubes, there is required only such equipment as is found in the home.

For making maple cream or "butter" there is no one percentage of invert sugar concentration that will be optimum under all conditions and for all recipes. Usually the best results are obtained if the sirup contains not more than 2% of invert sugar.

Apparatus:

- 1 transparent measuring cup
- 1 quart measure (milk bottle or fruit jar can be used)
- 1 container for mixing (sap-gathering pail, 16 to 20 qt. size)
- 1 long handled spoon or paddle
- 5 clean, dry, 4-oz. tumblers
- 1 bottle of "Clinitest"^a tablets with color chart
- 6 test tubes (approx. ½ inch diameter)
- 1 test tube holder*
- 6 medicine droppers (for use with "Clinitest" tablet)

* Made from a wooden block approximately 1 x 3 x 8 in. with a line of 5 holes, ⅝ in. in diameter and ½ in. deep. Label the holes in order, one for each of the dilutions.

Before taking a sample, the sirup must be well stirred so that it will be uniform in composition. The size of the sirup sample taken for this analysis is arbitrary; however, the sample must be measured accurately. In this outline the use of a cup for the sample size is suggested because it can be measured with the accuracy required for the test, although any other convenient unit may be used. To conduct the test, the sirup sample is diluted to different volumes with water and the different dilutions tested until one is found which will give a negative test for invert sugar.

Making the dilutions. The test is based upon 5 dilutions by volume of the sirup: 1 + 12, 1 + 20, 1 + 32, 1 + 40, and 1 + 60. These are prepared as described below.

^a"Clinitest," manufactured by the Ames Co., Elkhart, Indiana. Mention of commercial items of this kind does not specify recommendation by the United States Department of Agriculture over others on the market.

The 1 + 12 dilution: Measure and pour 2 quarts of water into the mixing pail. Fill the measuring cup exactly to the 1-cup mark with the sirup and pour the measured sirup into the pail. Let the cup drain until the sirup is nearly all removed (sirup drops fall at the rate of one every 30 seconds). Measure a third quart of water and use this to rinse the remaining sirup from the cup. To do this, fill the cup with water, stir, and pour into the pail. Repeat rinsing the cup until the quart of water is completely used. All rinsings are added to the dilute sirup in the pail. The pail should now contain a mixture of one cup of sirup plus 3 quarts of water. Now stir the mixture with the spoon or paddle until it is thoroughly mixed. This gives a 1 + 12 dilution (by volume) of the sirup. Using a clean, dry, 4-ounce tumbler, dip into the dilute sirup and withdraw a half-full tumbler (approximately 2 ounces). This is labeled 1 + 12 and is set aside for later use in making the invert test.

The 1 + 20 dilution: This is made by adding 2 measured quarts of water to the 1 + 12 dilution of sirup already in the pail. After stirring and mixing, using another clean 4-ounce tumbler, remove a 2-ounce sample and label it 1 + 20. Save this for use in the invert test described below.

The 1 + 32 dilution: This is made by adding 3 measured quarts of water to the 1 + 20 dilution of sirup already in the pail. After stirring and mixing, again remove a 2-ounce sample into a clean tumbler, label it 1 + 32, and save for testing.

The 1 + 40 dilution: This is made by adding 2 measured quarts of water to the 1 + 32 dilution of sirup already in the pail. After stirring and mixing, again remove a 2-ounce sample into a clean tumbler, label 1 + 40 and save for testing.

The 1 + 60 dilution: This is made by adding 5 measured quarts of water to the 1 + 40 dilution of sirup already in the pail. After stirring and mixing, again remove a 2-ounce sample into a clean tumbler, label 1 + 60 and save for testing.

Invert Test: Using the samples of diluted sirup prepared above, make the analysis for invert sugar, according to the following directions which are similar to those supplied with the "Clinitest" tablets.

Directions: Place 5 of the test tubes in the test tube block. Fill a clean, dry medicine dropper with the dilute sirup from tumbler labeled 1 + 60. Holding this dropper in an upright position, place 5 drops of this diluted sirup into the test tube located in hole 1 + 60. Using separate clean medicine droppers, in the same manner add 5 drops of each of the remaining 4 samples of diluted sirup to their corresponding test tubes. With a clean medicine dropper add 10 drops of water to each of the 5 test tubes. Remove 5 of the "Clinitest" tablets from bottle onto a clean piece of paper, and then place a tablet into each test tube in order, starting with test tube number 1 + 60. In dissolving, the tablets cause the contents of the tubes to boil. Do not move the test tube while the solution is boiling. Wait 15 seconds after boiling has ceased and then, starting with tube number 1 + 60, fill each test tube, in order, 2/3 full with water. Compare the colors in the test tubes with the first 2 colors of the color scale (which are labeled trace and +) supplied with the "Clinitest" tablets.

INTERPRETATION OF TEST

If the color developed in any of the test tubes is the same or bluer than the first color of the "Clinitest" color scale (trace), it indicates a negative test. If the color is a yellow-green, matching the second color on the "Clinitest" color scale (+), the test is positive. If the color produced is more yellow, or has shades of orange through brown, the test is strongly positive. If the color does not match either of these 2 colors, but is intermediate, the test is considered doubtful, \pm . The percentage values given on the "Clinitest" color scale have no relationship to this test for invert sugar in sirup and therefore are to be disregarded.

Examine the tubes in a room illuminated with a tungsten bulb, and with the aid of the first 2 colors of the "Clinitest" color scale record in a

vertical column a plus (+), minus (-), or doubtful (\pm) for each of the 5 tubes. Then compare this column with those found under the letters in Table 1. Under that column which has markings similar to those obtained in the test are given the minimum, maximum, or probable percentage of invert sugar in the sample tested.

Example 1: If all of the tubes are negative (sample A), then the sirup being tested contained less than 2% invert sugar.

Example 2: If the tube marked 1 + 12 is positive and all the others (1 + 20, 1 + 32, 1 + 40, and 1 + 60) are all negative, sample B, then the test shows that the sirup being tested contained at least 2% but less than 3% invert sugar.

Example 3: If the 1 + 12 dilution is positive, the 1 + 20 dilution is doubtful (positive or negative), but the 1 + 32 dilution is negative, sample C, then the test shows that the sirup contained more than 2% invert sugar but less than 4%, with the probability that the true figure is near 3% or midway between.

Example 4: If, after examining the colors in the 5 test tubes, it is found that the tubes marked 1 + 12, 1 + 20, and 1 + 32 are all positive, and the tubes marked 1 + 40 and 1 + 60 are negative, sample F, this test shows that the sirup being tested contained at least 4% but less than 5% invert sugar.

Example 5: If the 1 + 40 dilution color is doubtful (positive or negative) but the 1 + 60 dilution is negative, sample G, then the test shows that the sirup contained more than 4% invert sugar but less than 6%, with the probability that the true figure is near 5% or midway between these values.

It is quite possible that the sirup being tested has less than 2% of invert sugar and all the dilutions will give negative color tests. In such cases, to determine the color of a positive test it may be desirable to add 3 drops of corn sirup to the 4-ounce tumbler containing the last dilution (1 + 60) and make another color test. Using 5 drops of this solution in a clean test tube, add 10 drops of water and a "Clinitest" tablet. This test will be positive because of the glucose, the reducing sugar contained in the corn sirup.

DISCUSSION

This simple method for the determination of invert sugar is based upon a test which will distinguish between the amounts of invert sugar in the test solution that will react with a "Clinitest" tablet to give either a positive or negative color. For proper functioning the concentrations of invert sugar that will give either a positive or negative test should be within narrow limits. These limits are determined by the colors which readily distinguish between a positive and negative test. The first 2 colors of the "Clinitest" color scale were chosen since they had the greatest difference in hue and were therefore the most easily distinguished. Using these colors, the limits were found to be between 0.10 and 0.15 mg. of invert sugar in the 5 drops taken for the color test. On this basis it was found that dilutions of 1 + 12, 1 + 20, 1 + 32, 1 + 40, and 1 + 60 (on a volume/volume basis) would effectively reduce the invert sugar concentration of sirups containing 1, 2, 3, 4, 5, and 6% invert sugar respectively, so that 5 drops from one of these dilutions will contain more than 0.15 mg., while from a succeeding dilution they will contain less than 0.10 mg. The positive and negative tests at the 5 dilutions for maple sirups containing 1 to 6% invert sugar are shown in Table 1. There will be sirups that will contain an amount of invert sugar which will give a doubtful color test since the

5 drops will contain an amount of invert sugar that is between these limits. In such cases the maximum percentage is indicated by the next larger dilution which gives a negative test.

It was found that the test cannot be applied to sirups containing 7% or more of invert sugar, because there is no combination of dilutions that will give a negative test for one percentage and a positive test for the next higher percentage. The dilution that will give an invert sugar content of less than 0.10 mg. (negative test) per the 5 drops of the test solution for a sirup containing 7% invert sugar will give less than 0.15 mg. of invert sugar (negative to doubtful test) for sirups containing 8 to 9%.

The validity of this simple test was confirmed by analyzing a series of 12 maple sirups whose invert sugar content was established by Schoorl's copper reduction method (1). These sirups contained 1.15 to 6.00% invert sugar. The results obtained are presented in Table 1.

TABLE 1
"Clinitest" color reactions for maple sirups containing different concentrations of invert sugar

Sample No.	A	B	C	D	E	F	G	H	I	J
Dilution (by volume)										
1 + 12.....	—	+	+	+	+	+	+	+	+	+
1 + 20.....	—	—	±	+	+	+	+	+	+	+
1 + 32.....	—	—	—	—	±	+	+	+	+	+
1 + 40.....	—	—	—	—	—	—	±	+	+	+
1 + 60.....	—	—	—	—	—	—	—	—	±	+
Per cent invert sugar by "Clinitest"										
More than.....	0	2	2	3	3	4	4	5	5	6
Less than.....	2	3	4	4	5	5	6	6	7	7 (or more)
Per cent invert sugar by chemical analysis										
	1.15	2.01	2.52	2.96	3.49	3.96	4.67	5.00	5.60	6.00

Legend:

- Indicates a negative color test.
- ± Indicates a doubtful color test.
- + Indicates a positive color test.

From these results it can be seen that sirups which had an invert sugar content that approached a whole number, yielded 2 adjacent dilutions which gave a positive and negative test that could be easily distinguished and made it possible to determine the percentage within a range of 1% (samples A, B, D, F, H, and J). Sirups whose invert sugar contents lay between whole percentages (samples C, E, G, and I) yielded a positive and negative test with dilutions that were not adjacent, but were separated by an intermediate dilution that gave a doubtful test. Because of this, the percentages could not be determined with assurance closer than $\pm 1\%$, the limits being marked by the dilutions yielding the positive and the negative test. However, it will be noted that in most cases the percentage of invert was near that indicated by the dilution giving the positive test.

Since these studies were carried out, two other reagents, Sheftel Urine Sugar Test Case^a and Benedict's Qualitative Solution,^d manufactured by

Eli Lilly and Company, Indianapolis, Indiana, came to our attention and were subsequently applied to this use. Trial runs have shown that the Sheftel Test was applicable; however, in all cases it gave a negative color reaction one dilution step earlier than the Clinitest. Thus, in order to apply Table 1 to the interpretation of the Sheftel Test, the values for the range of invert sugar must be shifted one column to the left. The Benedict's Solution was found to be a sensitive qualitative test for very low amounts of invert sugar.

LITERATURE CITED

1. BROWNE, C. A., AND ZERBAN, F. W., *Physical and Chemical Methods of Sugar Analysis*, 3rd ed., 1941, p. 981, John Wiley and Sons, New York.